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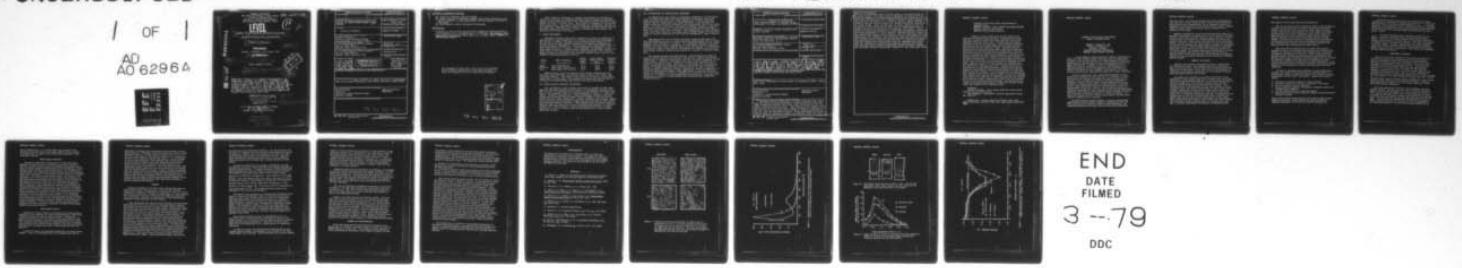
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CONTROL OF SHOCK-INDUCED POROSITY AND FAILURE BY PRECIPITATE-VA--ETC(U)
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The State University of New Jersey
College of Engineering
Bureau of Engineering Research
New Brunswick, New Jersey 08903

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BY PRECIPITATE-VACANCY INTERACTIONS.

10 Victor A. Greenhut

Final Report

Period: November, 1973--June, 1974

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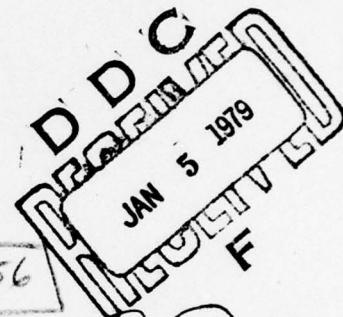
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"A Method of Controlling Shock-Induced Damage in Aluminum Alloys,"
A. M. Dietrich, V. A. Greenhut and S. K. Golaski, Proceedings, 1974 Army Science Conference, West Point, N. Y., June, 1974. Winner of second prize award.

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This report is intended to augment the results of research reported in "A Method of Controlling Shock-Induced Damage in Aluminum Alloys," which was presented at the 1974 Army Science Conference, West Point, New York, in June, 1974. This paper is included in the Conference Proceedings. The paper, representing the cooperative studies of the Ballistic Research Laboratories, Aberdeen Proving Ground, and the Materials Research Laboratory, Rutgers University, was awarded the second prize at this conference.

I. AGING TREATMENTS

Extensive tensile tests were conducted in order to establish an optimum set of heat treatments for further shock-loading specimens. All Al- $4\frac{1}{2}\%$ Cu tensile specimens were solution heat-treated at 525° C for 1½ hours and then rapidly quenched in icewater by employing a tilting furnace arrangement. All specimens were subsequently aged at room temperature for 48 hours. This treatment corresponds to the under-aged condition. Further heat treatments at elevated temperature were given to the critically and over-aged specimens. The optimum aging treatments, determined from Instron tensile tests, had the following average engineering stresses:

Aging	Heat Treatment	Yield Stress (ksi)	0.2% Plastic Offset Stress (ksi)	Ultimate Stress (ksi)
Under	Room temp > 48 hr	21.6	27.0	42.3
Critical	Room temp + 170° C / 17 hr	26.8	32.5	42.4
Over	Room temp + 220° C / 12½ hr	21.0	25.8	44.0

These results show the maximum yield stress obtained for the critical aging treatment and similar yield stresses for under- and over-aged samples. The tensile test results for various aging treatments and, in particular, the treatments given above can be used as criteria for shock-loading experiments and their evaluation.

II. SHOCK-LOADING SPECIMEN PREPARATION

Six cylindrical shock-loading specimens of Al- $4\frac{1}{2}\%$ Cu have been prepared and aged employing the three heat treatments outlined above. The two sets of specimens are presently being prepared for shock loading at two pressure levels by co-investigators at the U. S. Army Ballistic Research Laboratories. These specimens, together with the specimens previously shock-loaded (cited in paper), will be used to derive critical spall threshold, relative porosity and necking, change of precipitate and deformation morphology as functions of shock pressure and precipitate structure. Such quantitative information may be used as input for theories of pore nucleation and growth, to evaluate further the effectiveness of varied precipitate structure in spall suppression, and to develop engineering application of the vacancy gettering concept.

III. EXAMINATION OF SHOCK-LOADED SPECIMENS

In addition to the series of specimens referred to in the paper, a set of three specimens was shock-loaded at a pressure such that spall fracture did not occur. The examination of these specimens yielded results consistent with those reported. The incidence of large shock-induced pores increased with precipitate size in the order under-, critical, over-aged, and relative necking was also consistent with these observations. Significant precipitate growth was again found to result from shock loading. The consistency of these observations with those given in the paper lends further support to the mechanism of vacancy gettering as an effective mechanism of spall suppression.

SEM examination of sectioned specimens cited in the paper was also undertaken in order to extend porosity measurements and to reveal possible bulk diffusion of copper. The irregular nature of the spark-machined surface made it impossible to distinguish reliably pores of smaller size than those observed by optical microscopy. Determination of relative copper concentration throughout the sample by non-dispersive energy analysis proved inconclusive as a result of section roughness.

The specimens were polished with $\frac{1}{4} \mu$ diamond paste in order to yield a smooth surface and preserve fine pores. Optical microscopy examination of porosity showed that over-all porosity increased in the order under-, critical, over-aged, consistent with observations reported in the paper cited. Moreover, it was noted that the finer-sized, metastable precipitates proved most effective in reducing the number of larger pores and pore-joining related to spall fracture. The pores showed octahedral shapes, indicating vacancy coalescence on densely packed growth planes. SEM examination of the polished sections is continuing in order to evaluate the distribution of fine porosity as a function of precipitate-vacancy gettering. Possible bulk diffusion of copper is being determined, employing non-dispersive energy analysis.

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ence of the reflected tensile pulse forming pores which then act as crack nuclei. To verify such a mechanism, an Al-4.5 wt % Cu alloy was examined for shock-loading response as a function of precipitate structure which was controlled by prior heat treatment. The growth of metastable G. P. zones to stable θ -phase precipitates in such an alloy proceeds via vacancy-controlled diffusion of copper atoms. Thus, in the case of shock loading it was expected that such precipitates might act as "vacancy-getters" interfering with the coalescence of vacancies into pores. Transmission electron microscopy of an under-aged precipitate structure, prior and subsequent to shock loading, revealed distinct growth of the precipitates during shock loading. For an under-aged and over-aged alloy heat treated to yield similar tensile properties it was anticipated that although both alloys had the same yield strength, the over-aged alloy would be less effective in controlling spall fracture because the precipitates would be comparatively stable. This was confirmed by optical and scanning electron microscopy. The over-aged alloy showed considerably higher porosity, necking and incipient failure cracks when compared to the under-aged alloy.

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A Method of Controlling Shock Induced Damage in
Aluminum Alloys

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Studies of shock loaded metal crystals indicate that vacancies are produced during passage of a compressive wave and that these vacancies coalesce into pores under the action of the subsequent reflected tensile wave. Such pores are the primary nucleation sites for spall fractures. This mechanism is inferred from the fact that an extremely high density of tangled dislocations results from the compressive shock. Vacancies formed by intersection of these dislocations migrate and coalesce under the influence of the reflected tensile pulse forming pores which then act as crack nuclei. To verify such a mechanism, an Al-4.5wt% Cu alloy was examined for shock-loading response as a function of precipitate structure which was controlled by prior heat treatment. The growth of metastable G. P. zones to stable θ -phase precipitates in such an alloy proceeds via vacancy-controlled diffusion of copper atoms. Thus, in the case of shock loading it was expected that such precipitates might act as "vacancy-getters" interfering with the coalescence of vacancies into pores. Transmission electron microscopy of an under-aged precipitate structure, prior and subsequent to shock loading, revealed distinct growth of the precipitates during shock loading. For an under-aged and over-aged alloy heat treated to yield similar tensile properties it was anticipated that although both alloys had the same yield strength, the over-aged alloy would be less effective in controlling spall fracture because the precipitates would be comparatively stable. This was confirmed by optical and scanning electron microscopy. The over-aged alloy showed considerably higher porosity, necking and incipient failure cracks when compared to the under-aged alloy.

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A METHOD OF CONTROLLING SHOCK INDUCED
DAMAGE IN ALUMINUM ALLOYS

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In the design of armor to protect a vehicle, suppression of behind the armor fragments (spall resistance) can have significant influence on mission survivability [1]. These fragments increase the probabilities of incapacitating the crew, initiating stowed ammunition, and/or igniting fuel. Although a variety of approaches such as additional ballistic shielding, the use of liner materials or the compartmentalization of fuel and ammunition can be considered to mitigate the effects of spall fragments, much remains to be understood in relating basic armor material properties to spall resistance.

It will be shown that, once a microscopic mechanism is postulated for the occurrence of spall fracture, it is possible to tailor the response of a material to suppress the spall fracture process. Further, it will be demonstrated that the concepts introduced in designing a material are not simply research peculiarities but may be used in a building block fashion to improve material performance across a broad spectrum by tuning the microstructure to react in a desired manner to ballistic loading. Such an approach represents a significant departure from other approaches where conventional material properties like hardness, yield strength, tensile strength, ductility etc. are used as correlating variables to ballistic response. To answer the question of how to inhibit spall fracture, one must first outline a mechanism of its occurrence.

The microstructural damage induced in a metal or alloy during shock loading involves several fundamental mechanisms of flow, and while these are not necessarily unique to shock deformation, the resulting substructures are quite distinct from those arising from

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cold work [2,3]. The formation of mechanical twins and dislocation cell structures of small misorientation, as well as the occurrence of diffusionless phase transformations are all documented examples of fundamental flow mechanisms by which metals react to shocks. Another common structural feature is porosity, which is central to the remainder of this paper.

Numerous investigators [4,5,6] have observed the formation of a distribution of pores in a variety of sizes in the region of the spall fracture. These pores have been shown to occur in the zone of interaction between an unloading wave traveling behind the initial compressive shock and a tensile shock reflected from a free (unstressed) surface. Further, it has been shown possible to treat pore formation as a process of nucleation and growth (NAG) in response to the tensile stress-time history. Porosity damage accumulates until a sufficient pore density is present to cause pore link up and the formation of a fracture surface. If porosity damage can be suppressed by restricting its nucleation and/or growth, the tendency of a material to fracture under a given shock condition can be reduced. The question then arises: how can material microstructures be selected to suppress porosity damage?

POROSITY AND FAILURE

Typically the nucleation and growth of pores is characterized by a viscous drag model. One interpretation of the viscous drag model is based on plastic flow resulting from dislocation motion and interaction to form pores. While such a view is quite credible, an alternative microstructural interpretation of the success of nucleation and growth relations can be made.

The authors and co-workers [2,3] during observations of the morphology of shock loading copper single crystals have noted three interrelated microstructural features: an extremely high dislocation density, arranged in a fine cellular structure; local regions of extensive microtwinning; and distributed fine micropores. One striking feature of the micropores in some cubic crystals is their octahedral shape, with facets corresponding to (111) crystal planes. A similar observation has been made by Stevens and co-workers in examining shock-deformed aluminum [6]. They explain their observation with a dislocation interaction mechanism. An alternative view proposed here is that such crystallographic facets represent pore growth faces resulting from vacancy accumulation on (111) planes. Such a mechanism has been shown to cause vacancy loop and/or tetrahedral faulting of rapidly quenched metals. It remains to be shown that an extremely high density of vacancies is produced and that

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they migrate to form voids during shock deformation.

The intersection of mobile dislocations leads to the formation of vacancies via jog formation. The extreme density of tangled dislocations, observed as a major component of shock loaded structure, is thus consonant with the formation of a large number of vacancies during passage of an initial compressive pulse. Stress-aided diffusion of vacancies may be expected to enhance vacancy migration rates dramatically at the stress levels characteristic of shock loading. The high tensile stresses resulting from the intersection of the reflected tensile wave and the unloading wave should favor the accumulation of vacancies; and, as previously indicated, the pores may well adopt crystallographic growth facets.

Many metallurgical phenomena such as crystal grain growth, precipitate growth, and stacking fault formation are described theoretically by nucleation and growth laws dependent upon the energies of formation and migration of vacancies. Such vacancy-controlled phenomena usually involve elevated temperature, considerable times and low stresses when contrasted with shock loading times and stresses. An "enhanced" diffusion rate might well be expected at the high stresses and temperatures characteristic of shock deformation. It is thus postulated that pore nucleation and growth in the case of dynamic fracture can be attributed to a stress assisted vacancy diffusion mechanism.

In order to investigate this hypothesis, it was decided that a suitable test required the introduction of a variable factor in a metallurgical system to control the diffusion of vacancies and their accumulation at local sites to form pores. Ideally such a factor should:

1. function as an internal marker of vacancy activity;
2. distribute vacancies as uniformly as possible in the metal;
3. act as a potential sink for vacancies;
4. compete with pore formation and growth by occupying vacancies in a "competitive" diffusion;
5. vary its effectiveness in a controlled manner;
6. demonstrate control of pore formation, growth, joining and resulting spall fracture.

While several possible microstructural and macrostructural systems suggested themselves, a most suitable one could be invoked by applying developments in the field of nuclear technology [7].

Irradiation of metals may lead to the displacement of atoms resulting in the formation of vacancies and interstitials. Such point defects diffuse in the metal to form undesirable voids. It has been found that the introduction of fine, uniformly distributed, metastable precipitates which grow by the mechanism of vacancy diffusion and act as vacancy sinks are an effective means of controlling void formation [7,8,9].

A metal system containing fine, metastable precipitates was examined in order to determine whether such precipitates would function effectively as "vacancy getters" during shock loading and thereby control porosity and spall fracture. They were also employed as internal markers to examine the function of a vacancy mechanism in dynamic loading. Moreover, because they model many commercial alloys they indicate possible immediate applications of the vacancy getter concept to control shock failure. The results and conclusions of these experiments follow.

EXPERIMENTAL PROCEDURE

Al-4 1/2 wt.% Cu was selected as the material for testing. By selection of suitable heat treatments prior to shock-loading a variety of homogeneous precipitate distributions with differing potential as vacancy getters may be obtained. The alloy has been the subject of extensive investigation [10,11]. The precipitate structures and static properties have been well characterized. Moreover, it is a simple binary alloy model for many important commercial aluminum alloys and is typical of a wide range of other aluminum and non-ferrous alloys. The alloy shows a precipitate structure which varies from metastable copper-rich G.P. zones, coherent θ'' , semi-coherent θ' to stable incoherent θ (Al₂Cu) upon heat treatment.

The precipitates grow at elevated and room temperatures to the various metastable forms dependent on the diffusion of copper. Moreover, the precipitates are thought to act as vacancy traps due to favorable solute-vacancy interaction.

The alloy, fabricated as Instron tensile specimens and shock loading specimens, was heat treated to three conditions designated under-, critical- and over-aged. The alloy was solution treated at 525°C and step quenched (170°C/1 min) to room temperature in order to homogenize the precipitate structure and minimize the precipitate size. The alloy was then held at room temperature for more than 48 hours. This resulted in a homogeneous G.P. precipitate structure with a tensile yield stress of 1.31 MPa (19 ksi) corresponding to the under-aged condition. The alloy was then aged 170°C/8 hr. to

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grow θ'' precipitates, i.e. critical-aged, with a maximum yield strength of 1.86 MPa (27 ksi) and finally 300°C/4 1/4 hr. to grow θ' precipitates, over-aged, with a yield stress equivalent to the under-aged condition.

SHOCK LOADING EXPERIMENT

The general experimental configuration is that described in reference [4] and consists of an explosive plane wave lens (TNT, COMP-B) a cylindrical buffer block to attenuate the explosive shock, and a cylindrical sample surround block with the 25.4mm long x 12.7mm diameter sample inserted in the surround block. All interfaces are hand polished. The compressive shock wave generated at the explosive/buffer block interface is reflected from the free surface as a tensile shock wave. The samples are recovered in water. An initial set of experiments was run to determine at what buffer block thickness the over-aged sample would exhibit complete fracture. This thickness was determined to be 85.7mm (3.375 in). The under-aged and critical-aged specimens were then tested for this same set of conditions in order to provide a rapid, preliminary evaluation of the efficacy of the proposed damage suppression concept. As described in subsequent sections of this paper, the anticipated damage levels can be ranked based on initial alloy conditions so that by contrasting the response of the remaining two conditions to the response of the over-aged condition a critical comparison could be made. It is recommended that future tests be conducted with loading geometries more amenable to one-dimensional analysis so that microstructural parameters can be related to damage nucleation and growth through a better documented stress-time history. The use of lower impulses than required for complete spall fracture should be employed to establish early damage nucleation and growth effects.

POST LOADING ANALYSIS

Subsequent to shock loading, the macroscopic and submicroscopic morphology of the specimens was characterized employing a combination of optical methods and electron optical methods. Transmission electron microscope (T.E.M.) observations were made employing a JEM 200KV electron microscope. The results of observation after loading were contrasted with observations of undeformed material, and comparisons were made of the shock loading response of the three precipitate distributions: under-aged (G.P. zone), critical-aged (θ'') and over-aged (θ').

A series of results was anticipated based upon the vacancy getter concept. First, it was expected that if an extremely high rate of

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generation of vacancies and stress-aided vacancy migration existed, there should be a significant growth of precipitates relative to the undeformed material. If this precipitate growth characterizes an effective vacancy getter, then porosity should be most inhibited in the case of the under-aged material and porosity should be maximum in the over-aged condition. This phenomenon may be understood to result because the energy which drives precipitate growth arises from the reduction of the surface free energy relative to the volume free energy of precipitates. The finely dispersed G.P. zones (under-aged sample) should thus prove most effective in interfering with the vacancy migration to pores. Further, it might be expected that the inhibition of pore nucleation and growth should lead to the greatest suppression of porosity damage for the under-aged alloy in spite of the fact that the greater tensile yield strength of the critical-aged and higher work-hardening and ultimate strength of the critical- and over-aged specimens might imply more resistance to plastic deformation and failure.

RESULTS

Electron microscopy of the three alloy configurations prior to shock loading reveals that the under-, critical- and over-aged conditions correspond to homogeneously dispersed G.P. zone, θ'' , and θ' precipitates respectively. Because the under-aged, G.P. zone structure may be expected to yield the maximum relative change in precipitate size distribution, attention was concentrated on characterizing any observable change subsequent to shock loading.

T.E.M. micrographs were obtained from undeformed and shock-loaded under-aged material (in the vicinity of the spall fracture region). The observations were made with a similar $\bar{g}=[200]$ two-beam case with beam (zone) axis close to [001] for both samples. Typical bright field (B.F.) and dark field (D.F.) micrographs are shown in Figure 1. It may be noted from the B.F. and D.F. pairs that significantly larger precipitates can be distinguished subsequent to shock loading. In the shocked samples a high density of dislocations was also noted and many precipitates displayed dislocation loops surrounding the precipitates. The micrographs of Figure 1 were taken with conditions to maximize precipitate contrast, and thus these dislocation features are not readily apparent in the micrographs shown. The dislocation loops may be consonant with vacancy accumulation at the precipitate-matrix interface although the possibility of an unexpected dislocation-precipitate interaction under shock conditions cannot be ruled out. Attention should also be drawn to area P of the shock-loaded micrograph (B.F.). This light area may be noted to consist of several regular geometrical shapes. These areas remain bright when the foil

is tilted out of the diffracting condition. The size and traces of the areas at P together with the fact that their contrast must be due to absorption contrast lead to the conclusion that these are voids (or micropores) many of which are included in the foil. Many such areas are noted and indicate that pores grow by void coalescence. These observations are consistant with a vacancy mechanism of pore growth.

In order to characterize the changes in precipitate size and distribution critically, counts were made of the maximum dimension of the disc-shaped particles. A typical precipitate size-number distribution normalized to the total number of counts is shown in Figure 2. The undeformed, under-aged alloy shows a disk diameter maximum population at 80 \AA which corresponds to a zone thickness of less than 6 \AA [10]. Thus, the volume of this G.P. zone is less than $3 \times 10^4 \text{ \AA}^3$. During shock loading θ'' precipitates are formed and a θ'' precipitate maximum population was found at 280 \AA which corresponds to a zone thickness of approximately 15 \AA . The volume of this θ'' precipitate is thus approximately 10^6 \AA^3 . The maximum population particle has increased in size by more than 30 times!

The growth in the precipitate size during shock deformation must occur by a process of ripening. Thus, the steep rise in particle number at lower values of precipitate size is due to the fact that some precipitates grow at the expense of others (i.e. ripening). Since the growth of precipitates occurs by solute transport to the larger particles, vacancies must be occupied in compensatory reverse motion. This observation confirms the fact that large numbers of vacancies are created in shock loading with rapid migration rates. The vacancies may be expected to be generated by dislocation intersection, and vacancy-interstitial reaction; the rapid migration is probably stress aided.

These results demonstrate that the precipitates do act as internal markers of vacancy activity, distribute vacancies in the metal, and may act as a potential sink for vacancies (e.g. dislocation loops observed around precipitates). Moreover, it could be anticipated that the precipitates act as vacancy getters in competition with pores.

In order to evaluate the effectiveness of precipitates in controlling porosity damage, metallographic cross sections of the shock loaded samples were evaluated. These observations are shown by Figures

3 and 4. It may be noted that the porosity is least extensive in the under-aged specimen if an examination of the longitudinal extent of observable porosity above and below the spall zone is made. The over-aged specimen shows the maximum degree of porosity. These results indicate that the under-aged precipitate structure was most effective in controlling pore growth, while the over-aged structure was least effective. This is in agreement with the anticipated result that the most finely dispersed precipitates should act most efficiently as vacancy getters.

Relative necking was also measured as an indication of the degree of internal porosity generation and shock damage resistance. To some extent these results were obscured by the amount of plastic flow in late stages of fracture. The observations are summarized in Figure 5. It may be noted that the under-aged material showed less necking than the over-aged. This compares well with the porosity observations. However, the critical-aged alloy shows less necking than either of the other specimens.

This can be understood by examining the specimen cross-sections of Figure 3. The under-aged specimen has a major crack which has opened ductilly. This leads to more extensive necking compared to the critical-aged material in spite of the fact that porosity is lower in the under-aged specimen.

Figure 3 also shows the extent of failure control. The over-aged alloy has failed completely and shows further failure cracks which have resulted from the joining of pores. The critical-aged specimen shows almost complete propagation of spall failure across the specimen and indeed the crack has propagated to the surface at least halfway around the specimen. The under-aged specimen while showing opening of a major spall crack has considerable material integrity to the specimen surface. Thus, as anticipated the model of the precipitate as an effective vacancy getter and consequent failure control parameter appears viable. Design of shock resistant materials and material treatments to take advantage of vacancy gettering and porosity control appears to show an important potential.

SUMMARY AND RECOMMENDATIONS

It has been shown that the microstructure of a metal can be manipulated in a rational, systematic manner to control the level of damage induced by a shock wave. This control has been achieved by first postulating the mechanism by which damage is induced [in this

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case porosity damage modeled as resulting from vacancy coalescence during shock wave intersection] and then determining a microstructure [controlled precipitates] to compete for the vacancies and thus suppress damage. The particular alloy studied was selected to provide an uncomplicated model of the proposed process while retaining a major feature of commercial alloys. The experiments to-date indicate that:

- a. Vacancies are significant to the nucleation of porosity damage during shock loading as evidenced by strong precipitate growth in the under-aged alloy.
- b. At the shock strengths examined the potential for vacancy gettering action is more important to suppressing damage than are yield strength, ultimate strength, work hardening rate or ductility as shown by comparison of the results of the under-, over-, and critical-aged alloys.

This initial study has opened the door to a new concept in tailoring material response to ballistic loading. Two avenues can be fruitfully pursued. First, a detailed analysis of shock induced damage in vacancy gettering materials should be conducted to relate the control mechanism to the nucleation and growth parameters. Such a relation would assist in the interpretation of nucleation and growth sensitivity to fundamental material properties and perhaps, stimulate new ideas for control techniques. Secondly, while the alloy selected produces precipitates characteristic of those in the 2000 series of aluminum alloys, many other possibilities exist. Of special interest are the aluminum-zinc and aluminum-silicon binaries and the aluminum-zinc-silicon ternary. These alloys can also be used to model fundamental reactions occurring in commercial materials. Further, the aluminum-zinc-silicon system may be heat treated to produce both a vacancy getter phase (Al-Zn precipitate with maximum coherency analogous to the Al-Cu phase discussed above) and a dispersion hardening phase (incoherent Si particles finely dispersed to increase material shear strength but too small to act as crack nuclei). Since the relative particle sizes and distributions of these two precipitate phases can be controlled independently over a broad range, this alloy's structure can be manipulated to provide an optimum vacancy gettering action for spall fracture resistance without necessarily trading off shear strength.

It is hoped that, in addition to the possible applications of specifically developed alloys, a new approach can be established with respect to materials design and selection for ballistic loading: an approach based on tailoring material structure for a desired ballistic response.

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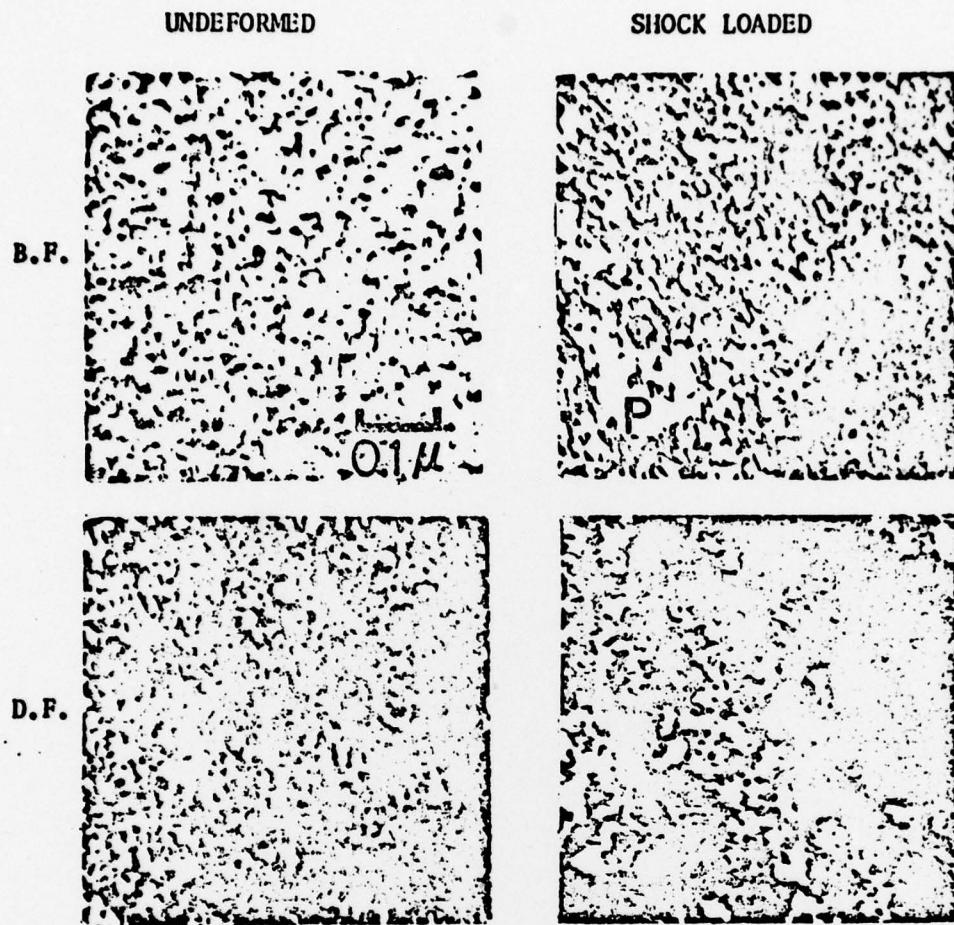


Figure 1. Transmission electron micrographs of samples from the under-aged Al-4 1/2 wt. % Cu alloy showing the same areas in bright field (B.F.) and dark field (D.F.), $\bar{g} = [200]$. All magnifications are identical. The shock loaded sample was taken from the area indicated by the arrow in Figure 3, approximately 2mm from the long axis.

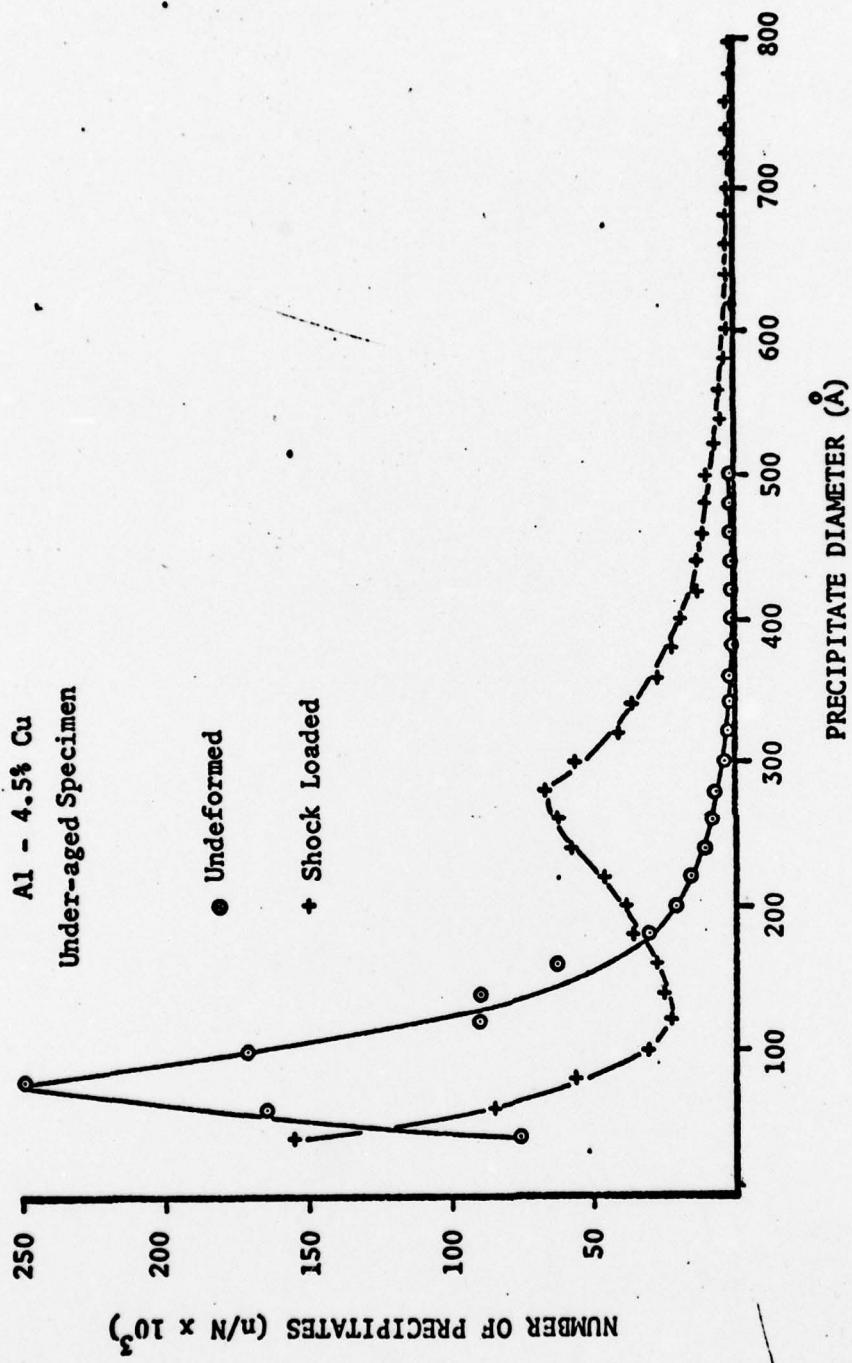


Figure 2. Normalized precipitate count indicating growth by ripening which occurs in the under-aged alloy during shock loading.

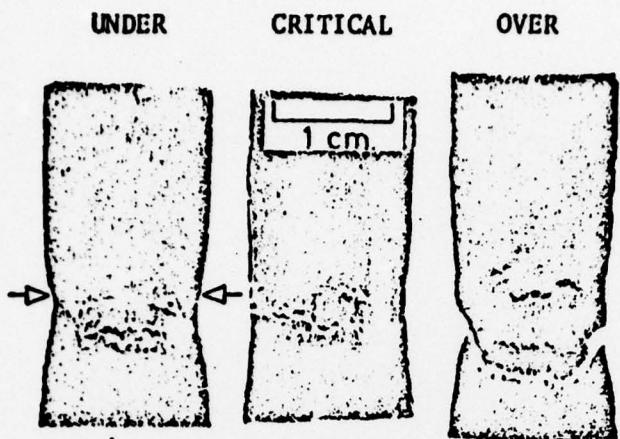


Figure 3. Macroscopic cross sections of under-, over-, and critical-aged samples after shock loading. The free surface of the specimen is the lower end shown in the figure.

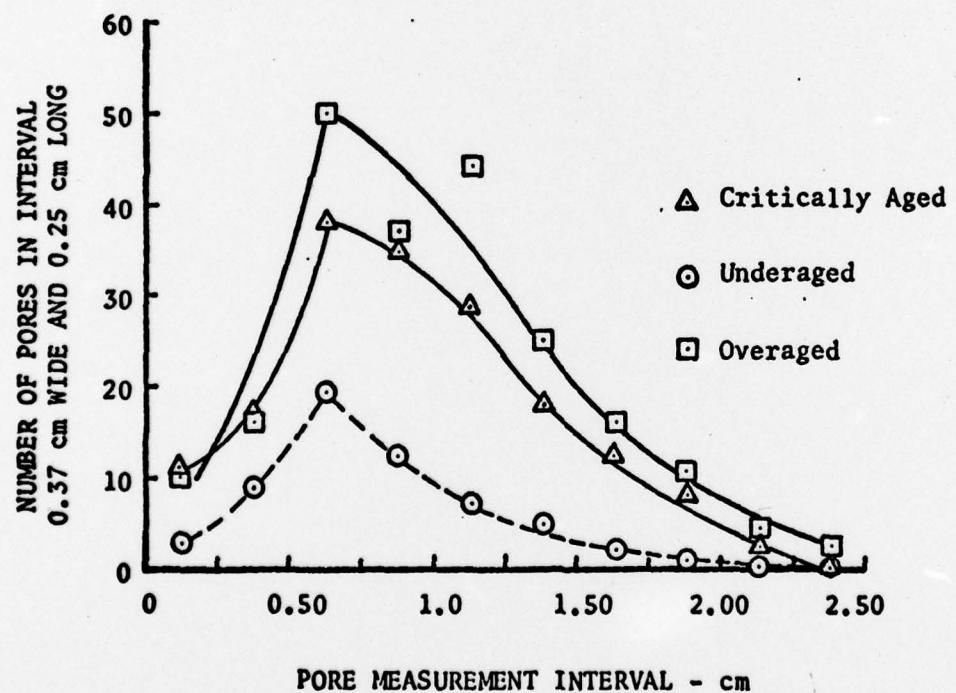


Figure 4. Number of pores in the 25 to 50 micron size range counted in areas 0.37 cm wide x 0.25 cm long versus counting area location, measured from the free surface.

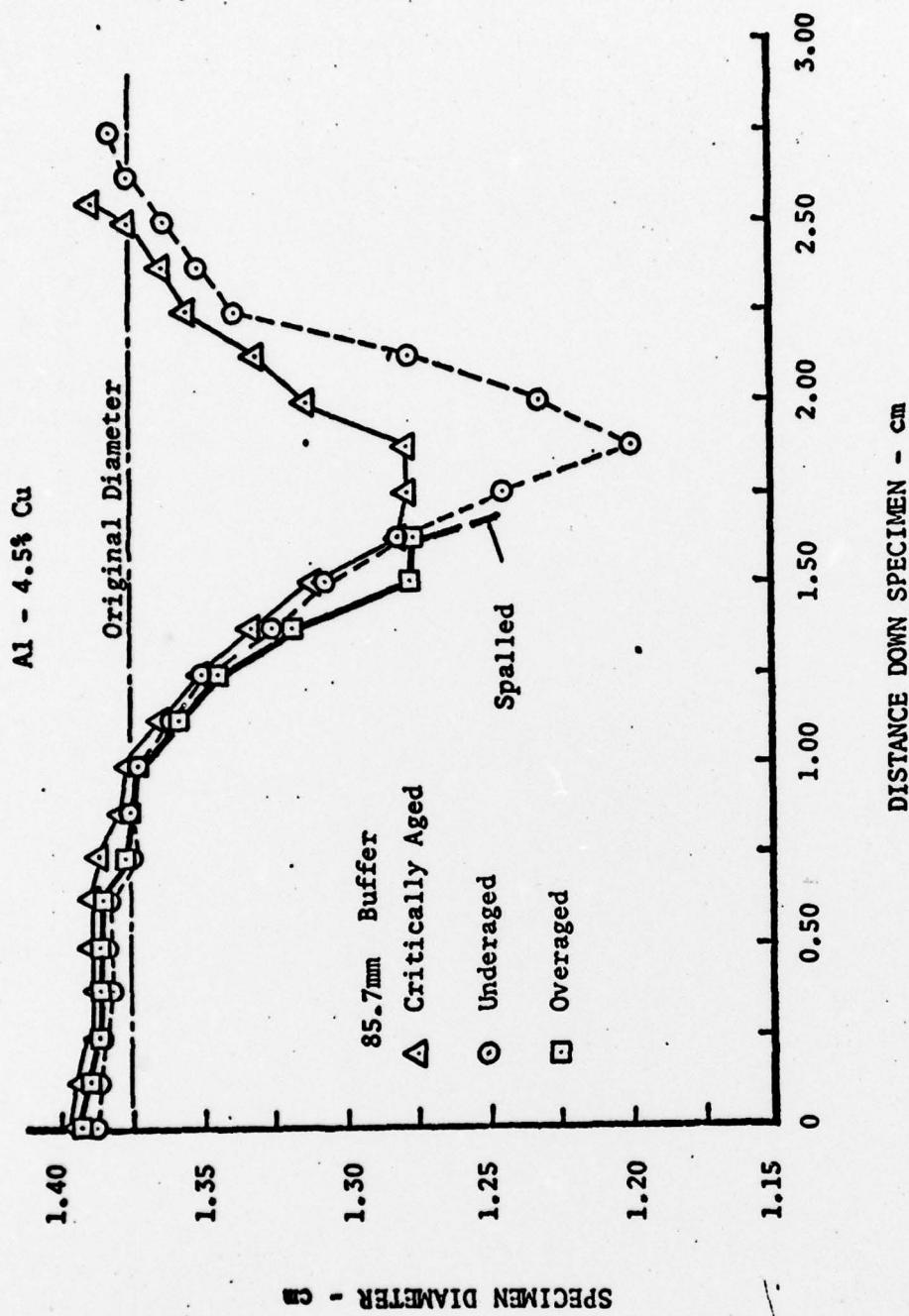


Figure 5. Necking response of shock loaded samples. Final diameter is plotted versus length from loaded end. Original diameter is shown by the dashed line.